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GUINIER-LENNE HIGH TEMPERATURE X-RAY CAMERA:

INSTRUCTIONS FOR USE

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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This manual was written to provide future users of Guinier-Lenne cameras in the Air Force Materials Laboratory with a clearly written set of instructions for set-up and operation of these cameras. This manual is intended to supplant the current manual, which is in poor condition and difficult to read. Borrowing information from the current manual, the Guinier-de Wolff operating instructions, and another manual, this author has attempted to gather as much relevant information as possible to instruct the reader in the operation of a Guinier-Lenne high temperature X-ray camera.

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11. (continued) Instructions for Use.

FOREWORD

This Interim Technical Report was prepared by the Universal Energy Systems, 4401 Dayton-Xenia Road, Dayton, OH 45432 under Air Force Contract No. F33615-82-C-5001/SB 5448-82-C-0076. It was administered under the direction of the Materials Laboratory, Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, OH with Dr. W. Wade Adams as the Project Scientist.

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1. INTRODUCTION

The Guinier-Lenne (G-L) camera is designed to photographically record wide angle x-ray diffraction (WAXD) patterns as a function of temperature. Structural changes can be followed in this way and temperatures of those changes determined. The types of processes typically examined are:

- thermal expansion,
- phase changes,
- chemical transformations, and
- recrystallization of amorphous materials;

but any change in a specimen which is temperature dependent and results in a change in its WAXD pattern can also be examined with this camera.

The G-L camera is capable of heating a sample from ambient conditions to 1200°C. The time to reach this or any other final temperature is fixed at intervals of 3 to 120 hours. The temperature may be increased or decreased during the experiment with the sample maintained at the final temperature or allowed to return to ambient at the end of the run.

The sample chamber may be evacuated or swept with a gas, either inert or reactive.

The film cassette is capable of recording diffraction angles from 3 to almost 90° 2θ, with a minimum $K\alpha_1$ and $K\alpha_2$ dispersion. The position of the film can be marked at 0° diffraction angle. The film cassette can be moved vertically by hand for a series of diffraction patterns or by electric motor for continously changing patterns. The film size is 174 mm by up to 260 mm. Film speeds can be varied discreetly from 0.5 to 20 mm/hr for maximum dynamic exposure times of 500 hours.

The width of the diffraction slit can be varied with factory prepared slits of 1, 3, and 5 mm. The wider a slit the more intensity reaches the recording film but with a corresponding decrease in time resolution.

2. CAMERA CONSTRUCTION

This camera, shown in Figure 1, is designed to be mounted horizontally with the focussing circle parallel to and 300-350 mm above the table. The support base has three legs, each with leveling nuts (14) to adjust the height and tilt of the focussing plane. One of the support legs is situated directly below the monochromator center so that the camera may be pivoted during the coarse adjustments while maintaining a constant take-off angle and anode-to-monochromator distance.

The upper base is attached to the lower base via a pivot controlled by the reflex wheel (13). Attached to the upper base are the monochromator housing (2), sample chamber (5) and furnace (6), and the film cassette holder (9) and slide (10). The electric film cassette motors to are located within the upper base.

The monochromator housing (2) contains the quartz monochromator crystal (16) (shown being inserted in Figure 2) and various adjusting controls (see Figure 3). The x-rays enter the housing through the attached metal bellows (1) (usually wrapped with lead foil) and exit into slot F1 (3) after being diffracted by the monochromator crystal.

The sample chamber and furnace are an integral unit through which the x-rays pass. X-rays enter through a beryllium window at slot F1, pass through the sample at the center of the furnace, and the main beam is stopped by the beam catcher (8) while the diffracted beam exits through another beryllium window and the diffraction slit (32). The diffracted rays are then recorded on the photographic film in the film cassette.

The heating block consists of a cylindrical metal housing containing various openings and holding a ceramic mass and outer cooling coils. The ceramic mass is wound with platinum-rhodium wire and is connected to the heater control, which electrically heats the ceramic mass and the sample as well. Cooling water (12) cools the outer surface of the metal housing preventing the film from getting hot. The vacuum port (4) can be connected to a vacuum pump or any sweep gas cylinder.

The sample holder (shown in detail in Figure 4) can hold films, foils, powders (pressed into a platinum screen or other holder, or any other sample approximately 10 mm x 5 mm. The head of the sample holder is made of a platinum-iridium alloy and connected to the screw base with a corundum tube. A platinum-rhodium/platinum thermocouple is imbedded in this tube. Samples can be loaded by removing the entire holder or through the side sample access port (7).

The beam catcher (8), located within the furnace assembly can be moved in and out of position electro-magnetically (35). The main beam can mark the film position by momentarily moving the beam catcher to the open position while the marking diaphragm (31) is in place (see Figures 5 and 6). The beam catcher must also be placed in the open position during certain phases of alignment.

The film cassette holder (Figure 7) is attached to the film cassette slide such that the recording film is coincident with the diffraction circle described later. The holder can be moved up or down by hand by squeezing the release handles (11). The motors, which can be electronically switched among several speeds, drive a shaft which in turn drives the slide holding the film cassette. The slide position can be observed on the film cassette reference scale (33).

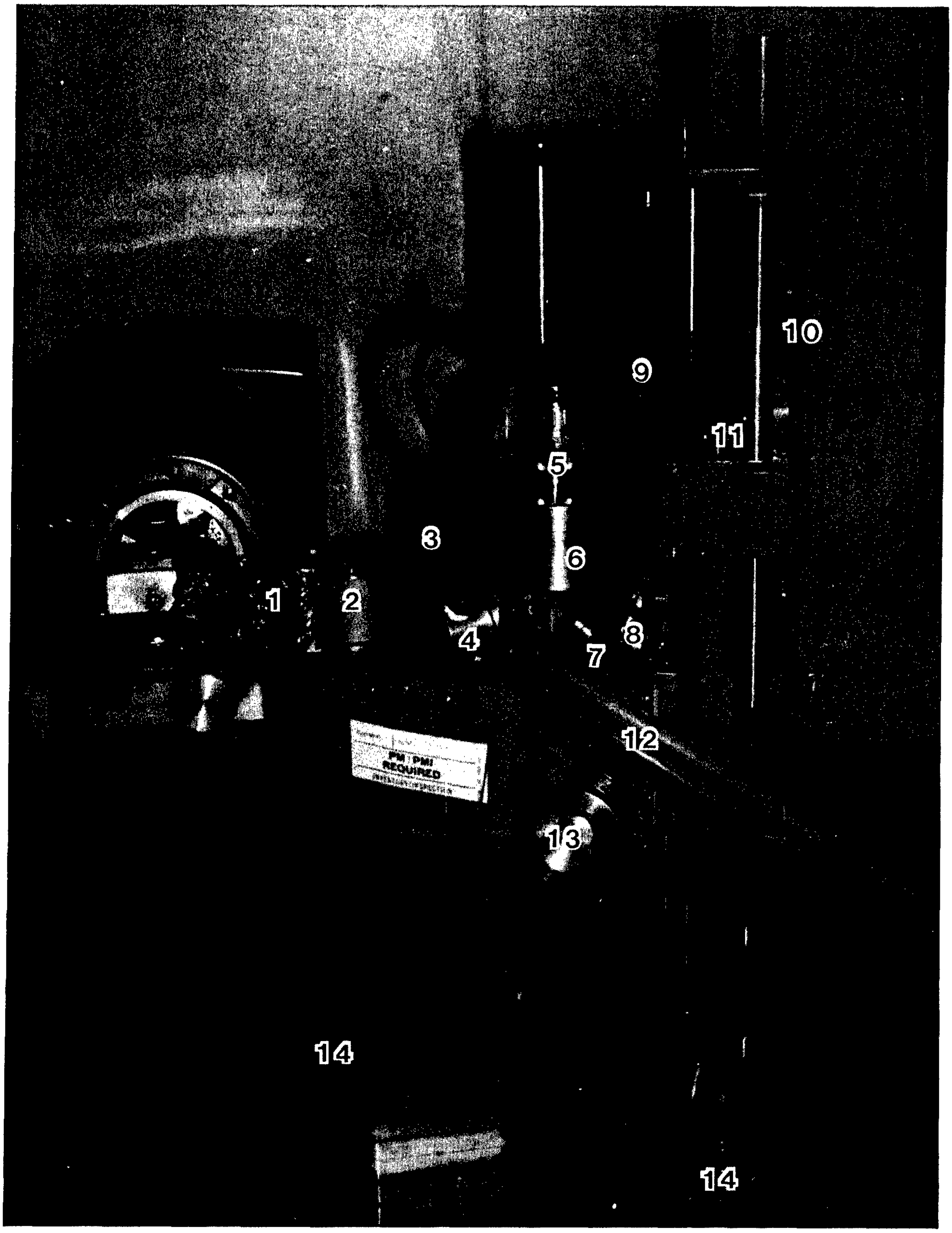


Figure 1. Guinier-Lenne Camera.

Figure 2. Monochromator Housing (front view).

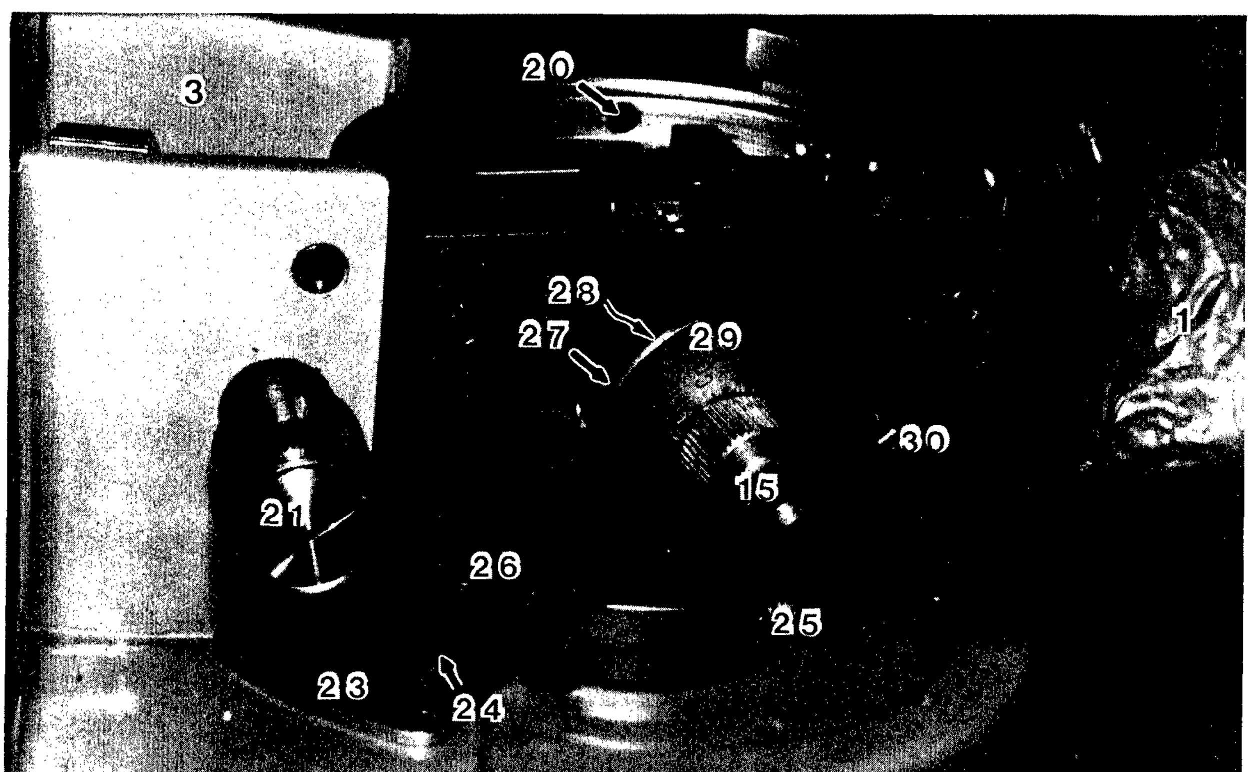


Figure 3. Monochromator Housing (back view).

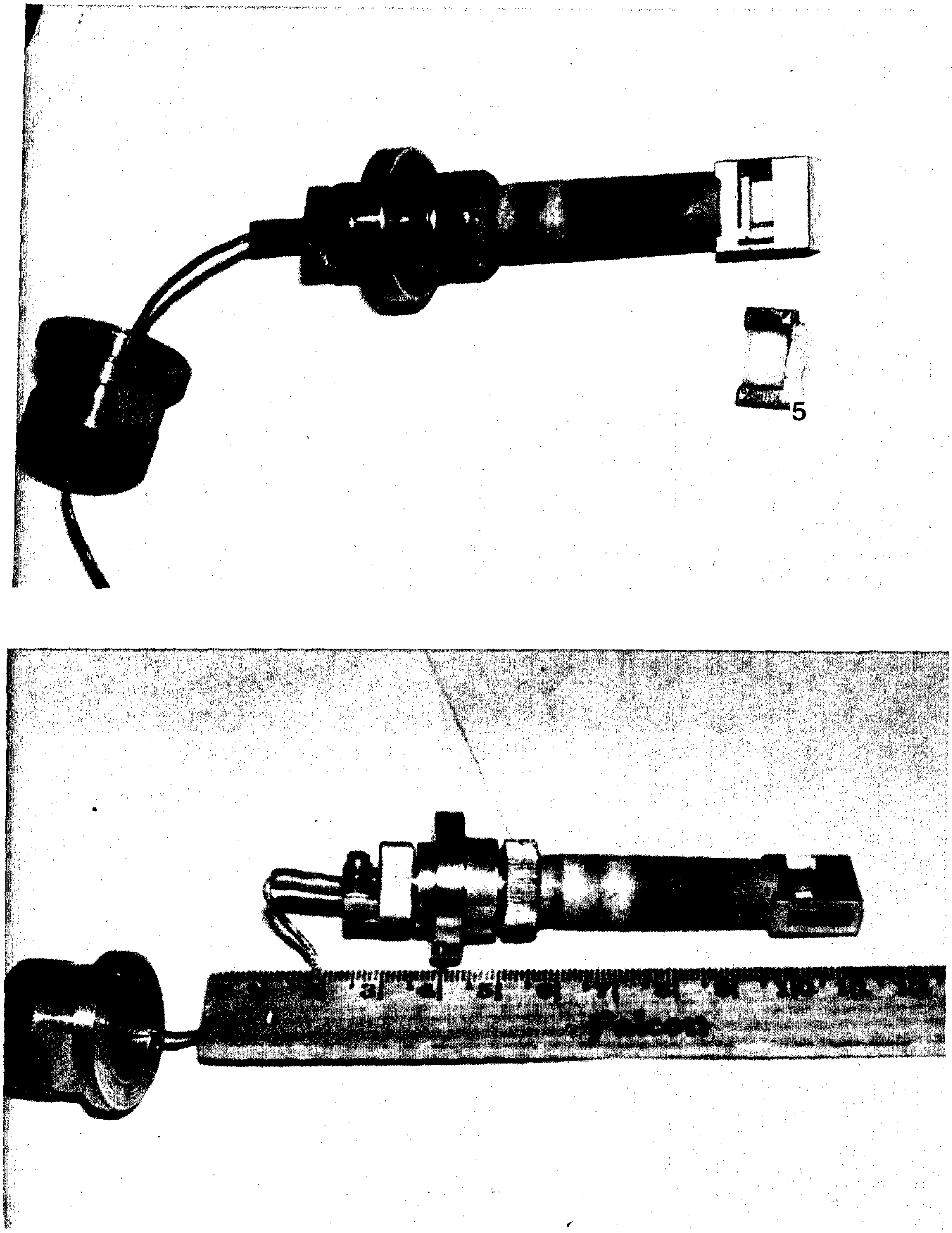


Figure 4. Sample Holder.

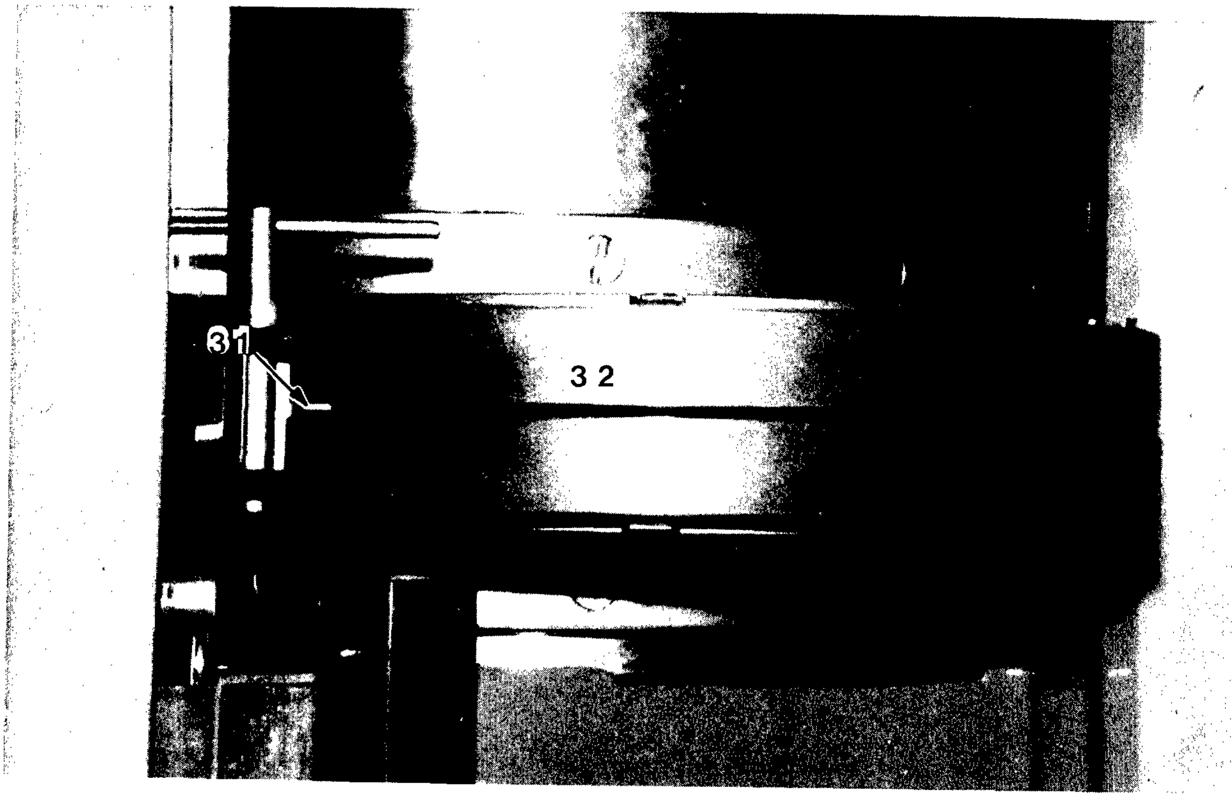


Figure 5. Scattering Slit Area.

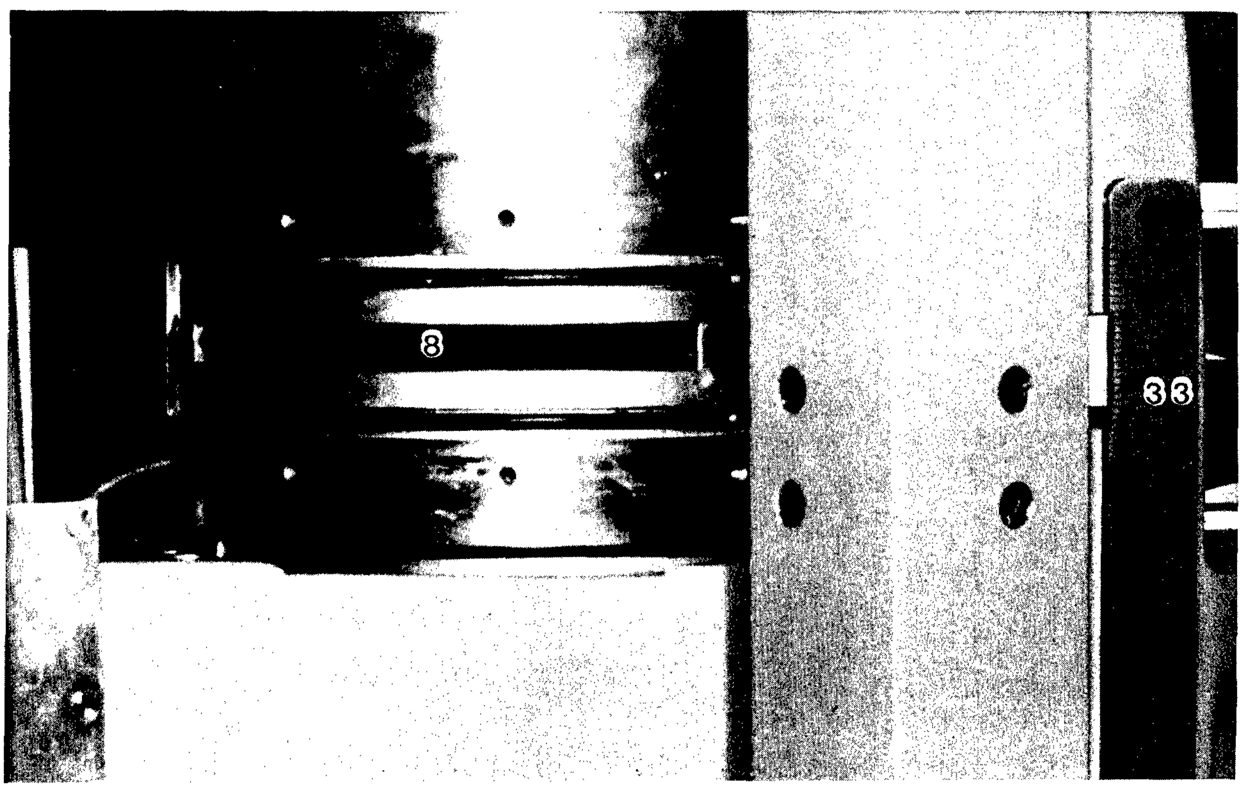


Figure 6. Scattering Slit Area - diffraction window removed.

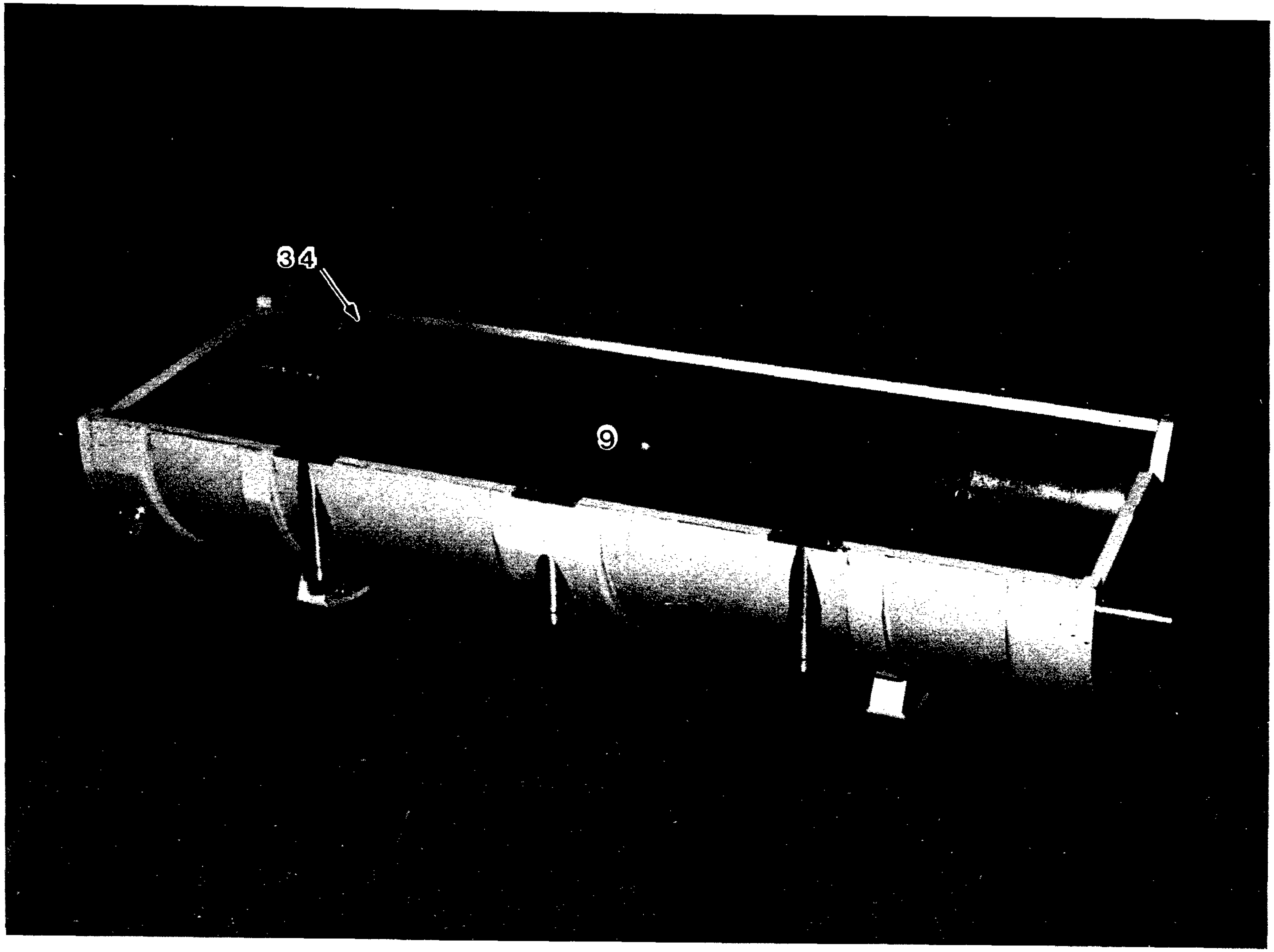


Figure 7. Film Cassette.

3. FOCUSSING PRINCIPLE

This camera is configured as shown in Figure 8. The main x-ray beam leaves the anode at a fixed take-off angle and is diffracted by the quartz monochromator (M) to the sample (S) which diffracts the beam onto the diffraction circle (DC). The anode to monochromator distance (L), incident angle (γ), and diffracted angle (2θ) vary for different wavelengths (see Table 1).

TABLE 1. Quartz Monochromator Settings

Anode Material (Radiation Type)	Wavelength K (nm)	L (mm)	γ (°)	Monochromator Angle (° 2θ)[a]
Copper (Cu)	0.15418	110	9	26.6
Cobalt (Co)	0.17902	120	11	31.1
Iron (Fe)	0.19373	130	13	33.7
Chromium (Cr)	0.22909	150	16	40.1
Molybdenum (Mo)[b]	0.07101	110	14	12.2

a) 2θ for the $10\bar{1}1$ Quartz reflection.

b) The first order molybdenum radiation involves the use of a special quartz crystal. Second order molybdenum radiation can be obtained approximately using the copper settings with the general quartz crystal.

The geometry of this camera is of the parafocussing type. This means the diffracted reflections of the sample converge to a line in space on the diffraction circle. The geometry, Guinier-Seemann-Bohlin, is the same as that used in the Guinier-de Wolff camera. The spread in diffraction lines due to the separation of $K\alpha_1$ and $K\alpha_2$ is minimized by the parafocussing and at $2\theta=29^{\circ}$ for copper radiation the separation is in fact zero.

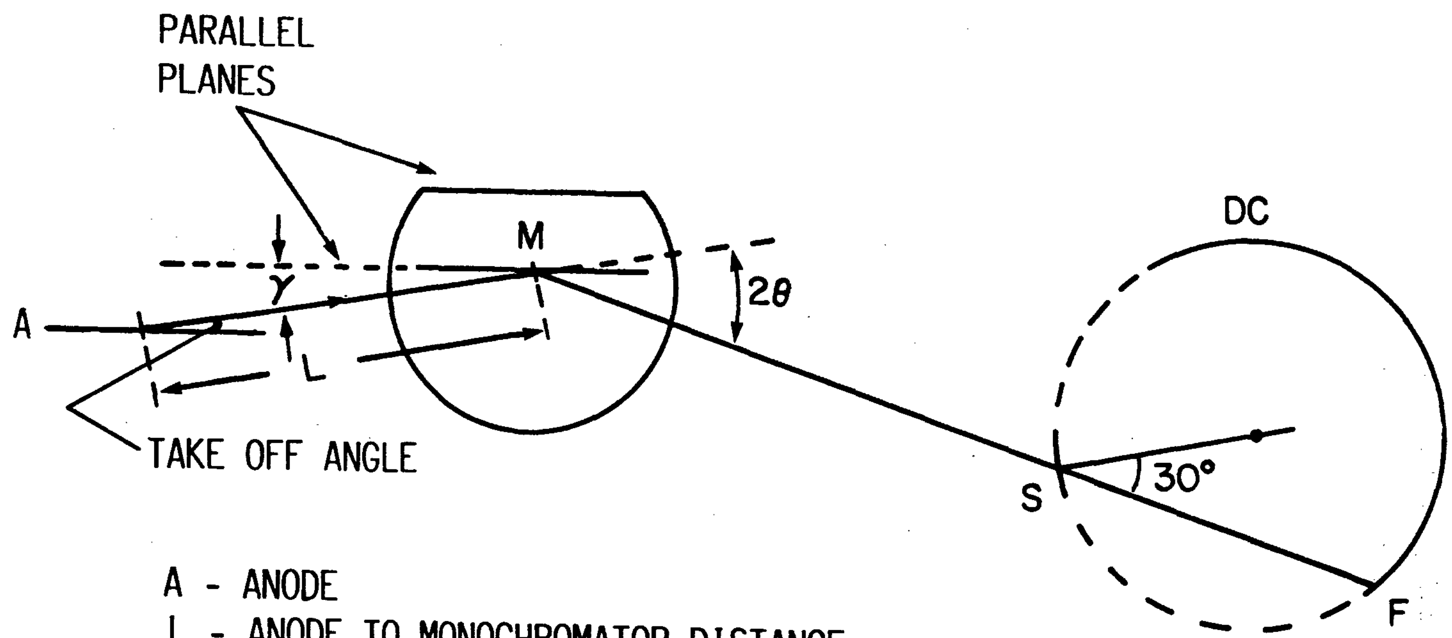
The quartz monochromator uses the $10\bar{1}1$ plane of quartz with the crystal surface cut at an angle $4\text{-}1/2^{\circ}$ to the $10\bar{1}1$ plane. This is shown in Figure 9. The exaggerated angles in this figure illustrate the diffraction detail occurring in the monochromator. The cut angle of $4\text{-}1/2^{\circ}$ to the $10\bar{1}1$ plane allows the beam to focus to a distance several times the monochromator-to-anode distance.

The quartz crystal is curved to obtain the parafocussing condition but not in a circular arc, rather in an arc along a logarithmic spiral originating on the anode focal spot. This allows the rays of finite divergence (aperture angle α) to strike the crystal all at the same angle. Rays originating from a single point when diffracted by this crystal do not focus at a point or line but rather envelop a caustic. This allows the camera to utilize the finite size of the anode focal spot located at the cross-over section which depends on the aperture (α) and the wavelength. For copper radiation and an aperture of 3° (full crystal width), the cross-over depth (t) is 0.3 mm (see Figure 10). A spot focus has a projected width of 1 mm allowing the use of the full aperture. Only a limited aperture can be used with a line focus due to its smaller projected width. However, since t is proportional to α^2, a greater part of the aperture can be used more effectively when using the line focus.

The location of the cross-over region is dependent on the position of the crystal used and therefore on the diaphragm (18) setting. This also controls the aperture. Since it is desired to use the center of the crystal, particularly for line focus, the optimum distance, L, also depends on the diaphragm setting.

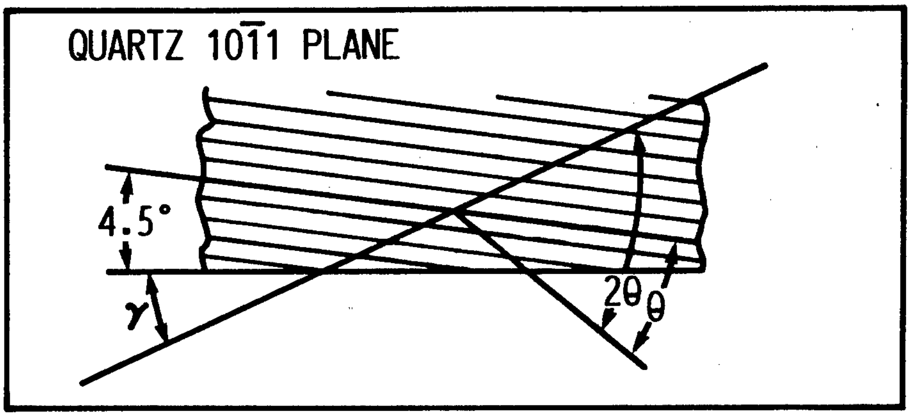
Only when L is set for the maximum useful aperture will it be correct for all apertures. This explains, to a limited degree, the interrelationship among the settings for L, take-off angle, and diaphragm in the Alignment Section (5).

The center of the diffraction circle is off-set from the main beam by 30° and defined by the focal spot (F) at 220 mm from the monochromator and a radius of 57 mm. The main beam passes through the sample which is located tangentially to the diffraction circle. The 57 mm radius diffraction circle makes a linear distance of 2 mm on the recording film equal to 1° 2θ.

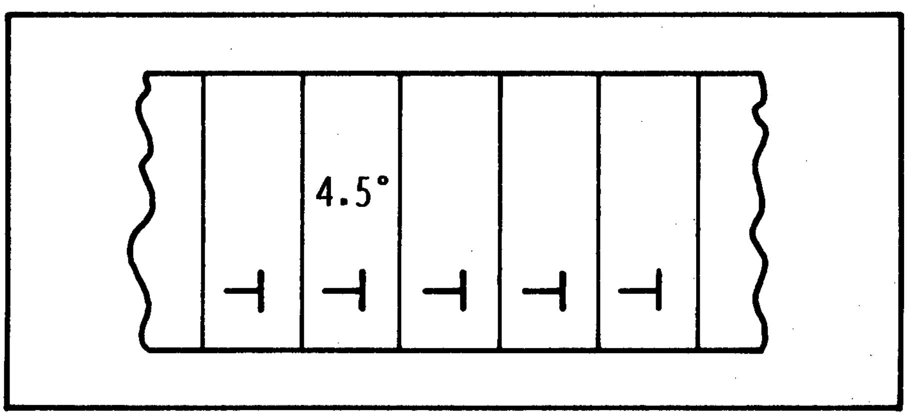


A - ANODE
L - ANODE TO MONOCHROMATOR DISTANCE
M - MONOCHROMATOR (16)
S - SAMPLE POSITION (5)
DC - DIFFRACTION CIRCLE
F - FOCAL SPOT
γ - INCIDENT ANGLE
2θ - DIFFRACTION ANGLE

Figure 8. Configuration of Camera.



FOR CuKα 2θ=26.6°



TRACE OF $10\bar{1}1$ ON SURFACE

Figure 9. Monochomator Detail.

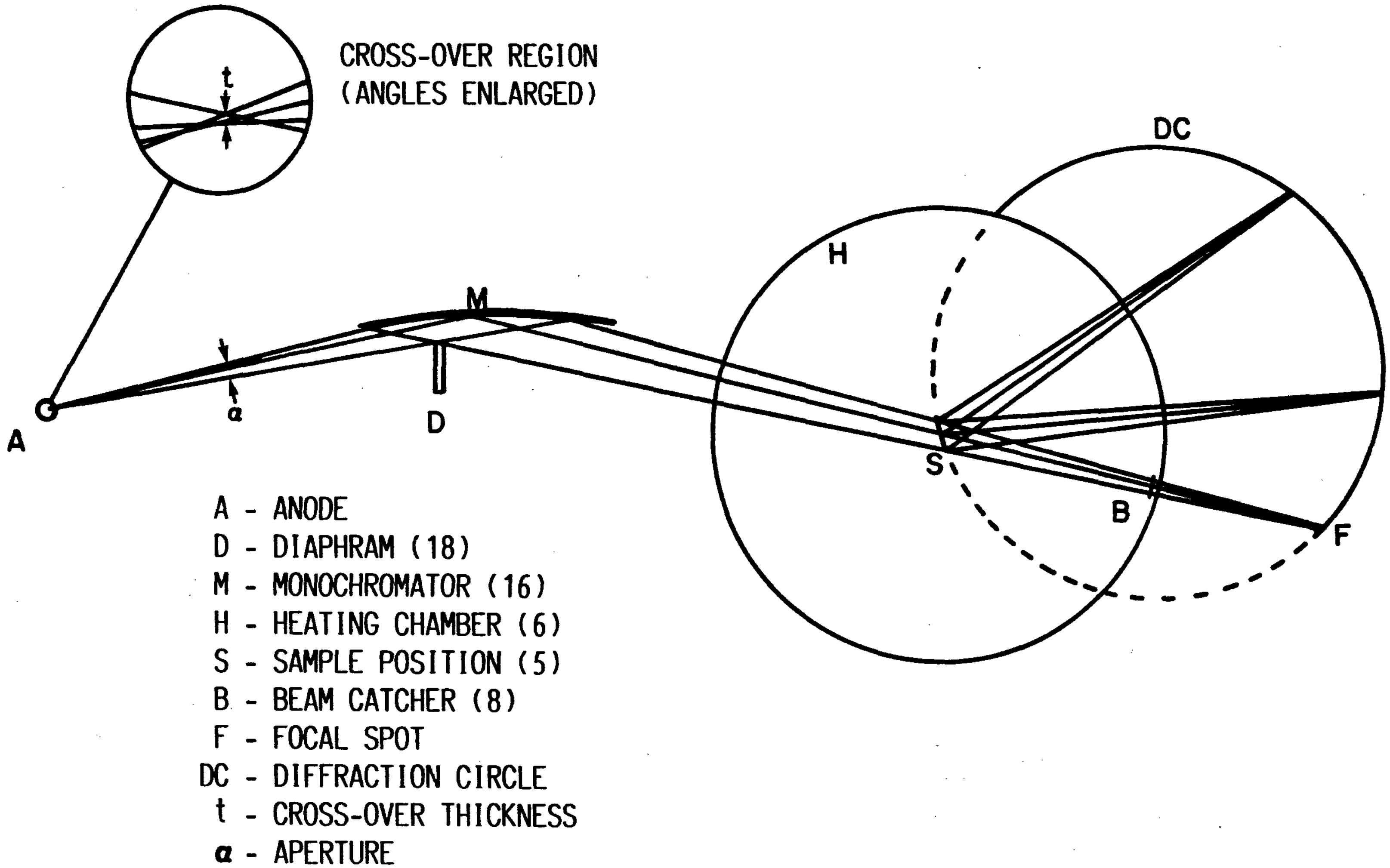


Figure 10. Focussing Principle.

4. SAFETY

The Guinier-Lenne camera, being an older instrument, has several safety problems associated with it which will be discussed in this section.

X-rays may be shut off by switching off the power to the tube or with port safety shutters if the tube is so equipped. The G-L camera in building 450 is set up on a General Electric XRD-5 generator which is fitted with Supper safety shutters. Wearing a x-ray film badge is required for operators of x-ray equipment and the use of a ring badge is recommended, particularly when aligning this instrument. The instrument should also be periodically surveyed for stray x-ray scattering.

The main x-ray beam was designed to pass through a metal bellows (1), which now has been found to occasionally leak x-rays, and through an open slot, F1 (3), after the monochromator. Both of these areas should be covered with lead foil. The foil over the slot also prevents anyone from inadvertently placing a finger in the beam path. During the normal operation of this camera, the x-rays should be shut off whenever the film cassette holder is removed. This holder not only holds the recording film but also blocks the diffracted rays and main beam. The main beam catcher also blocks the main beam but it is not interlocked to close if the cassette holder is removed. The main beam window in the cassette holder should be opened only during the alignment of the camera.

Several times during the alignment procedure, the main x-ray beam is open to the outside and accidental exposure to the operator is possible. The operator should be aware of where the main x-ray beam is coming from and where it is going while aligning the camera and avoid those areas. Particularly dangerous is the situation when the film cassette holder main beam window (34) is open. The operator may have to move in front of the film cassette to shut off power to the tube. He must first move the cassette holder vertically on its slide to block the beam before moving to the control panel. The fluorescent screen used to

observe the beam image in the alignment section can be attached to a stationary object or hand held when attached to a long (6 - 10 inch) handle of wood or plastic.

5. ALIGNMENT

5.1 Preliminary Adjustments

These adjustments are all made with the x-ray generator off.

5.1.1 Crystal Insertion - The quartz monochromator crystal (16) must be carefully placed in the monochromator housing (2) as shown in Figure 2. Note that the metal backing plate is on the side of the housing with the flat side and the hinged end of the crystal is pointed towards the side opposite the x-ray tube tower. The curvature dial (15) is then turned inward (clockwise) until the screw just touches the backing plate and then further until the scale (29) indicates the approximate curvature for the target anode material used.

5.1.2 Camera Position - The parafocussing geometry discussed earlier makes the positioning of the camera very important. The text of this manual uses the values of length and angles for copper radiation; if a different radiation is used then substitute the appropriate length and angle values from Table 1.

Determine the position of the anode target in the x-ray tube. For General Electric CA-7 or CA-8 tubes, the anode position is in a plane 1-1/32 inches from the very top of the cooling head and centrally located within the tube tower. Measure the take-off angle from that position and plane. Move the entire camera by hand so that the center of the monochromator housing is located at a take-off angle of 3 - 9^{o}. The larger the take-off angle, the greater the intensity and hence the easier alignment, but with less resolution.

The distance from the center of the anode to the center of the monochromator housing should be adjusted by moving the camera to L=110 mm. The flexible metal bellows should be clamped to the x-ray tube shutter for safe operation of this camera. The position of the camera housing foot beneath the monochromator housing should not be changed again until the Final Adjustments Section (5.2).

The height of the camera is adjusted using the leveling nuts (14) on the legs. The anode target, monochromator center, and film cassette beam window should all be at the same height in a plane parallel to the x-ray table.

Pivoting the camera, the angle between the x-ray anode target, monochromator center, and film cassette beam window should be fixed at the 2θ angle for the $10\bar{1}1$ quartz reflection of 26.6°. These and some of the other adjustments in this section can be done more easily by removing the furnace (6) by loosening the screws on top of the heating chamber and lifting it out of the way. The film cassette holder must be removed first to gain access to the furnace.

The angle between the flat side of the monochromator housing and the incident beam (γ) should be set at 9° using the monochromator micrometer (21). The indicator scale (23) on the outside of the monochromator housing should correspond approximately to the radiation type being used. The monochromator is factory set for copper so other radiation types require the housing to be changed.

To change the monochromator housing setting, loosen the lock screw (25) under the housing and manually turn the housing to the desired scale setting. Make sure the fine adjustment lever arm (22) rests against the micrometer screw before re-tightening the housing lock screw.

5.2 Final Adjustments

Most of these adjustments must be made with the x-rays on, however; turning off the generator or closing the port shutter may be required if additional gross adjustments are made.

First follow the instructions below under the Camera Housing Adjustments Section (5.2.1) with the x-ray tube set in the spot focus orientation. Reposition the x-ray tube in the line focus orientation and go on to the Crystal Monochromator Adjustments Section (5.2.2). Return to the beginning of the Camera Housing Adjustments Section (5.2.1) and repeat the instructions with the line focus until no further changes are needed. In practice only a few iterations are required.

5.2.1 Camera Housing Adjustments - Place a fluorescent screen just after the monochromator housing (position F1). Move the camera housing by hand without rotating it, to increase the take-off angle until the beam image is at its brightest position. If a shadow of the monochromator fine adjustment lever arm (22) is visible in the F1 image, then the monochromator housing must be rotated as described in the Camera Position Section (5.1.2). This can be accomplished without the need for other monochromator adjustments, but the x-rays must be turned off.

Turning the reflex wheel (13) will cause the image at F1 to move side to side if the quartz curvature was correctly set. Set the reflex wheel so that the main beam passes through the beryllium window as seen by the beam's image on a fluorescent screen at F1. The fluorescent screen, at F2, can be used to view the main beam image as it moves from side to side when the monochromator micrometer is turned (also known as turning through reflex). The F2 position is ~10 cm behind the film cassette beam window (34) which also contains the toothed screen. Remember the marking diaphragm (31) must be moved out of the way counter-clockwise, the film cassette placed in its lowest position, the room fully darkened, and the beam catcher (8) opened in order to see the main beam at position F2. A sheet of lead placed over the F1 area

reduces the scattered radiation reaching your hands while making these monochromator adjustments. The monochromator micrometer should be used to produce a main beam image of maximum intensity; changes in the reflex position may be required to maximize the image intensity.

If the distance L is correct, the image at F2 should appear symmetrically from the sides and disappear in the middle or vice versa when the camera is moved by the reflex wheel. If necessary, move the camera by hand, again without rotating it, to get the correct length L.

For a line focus, the image at F1 is a narrow band. Position the diaphragm (18) using handle (26) until it just cuts into the main beam image and no separate lines are visible. The diaphragm may have to be changed several times during the alignment.

If the image at F2 disappears top to bottom when the monochromator micrometer is changed, then the height of the camera is incorrect. Use the leveling nuts (14) to adjust the camera height until the problem is corrected, maintaining the optical plane parallel to the x-ray table. If the top to bottom movement of the image at F2 cannot be corrected with the leveling nuts, the problem is probably in the x-ray tube itself; i.e. the anode line focus is not vertical.

5.2.2 Crystal Monochromator Adjustments - The camera must be aligned well enough so that a fluorescent screen placed in the F2 position clearly shows the main beam.

Figure 11 shows the possible appearances of the beam image on the fluorescent screen at F2. A rectangle is the desired shape of the image. If the curvature of the quartz monochromator crystal is incorrect the image will appear as a trapezoid. Turning the curvature dial (15) slightly will vary the image from trapezoidal to rectangular to trapezoidal. When the dial setting produces a rectangle, lock the dial with its screw (28). A few trial exposures of the main beam should be done at this point; open the beam catcher at minimal power for a few seconds to expose film placed in the film cassette at several positions.

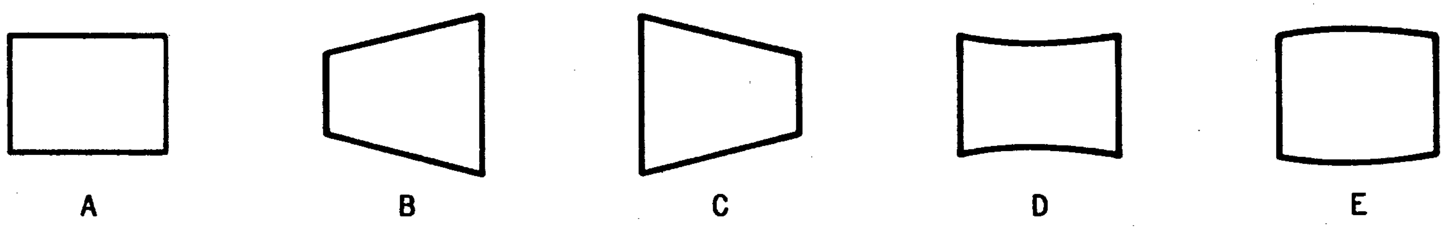
The $K\alpha$ doublet should be clearly visible as diagramed in Figure 12 if the curvature is correctly set.

The torsion on the quartz crystal should only have to be reset if several crystals are being used. A small screw (19) must be adjusted so that the image at F1 stays symmetric when the camera is moved through its reflex. Access to this screw is through a hole (20) in the monochromator housing. The monochromator housing must be closed when the torsion is adjusted.

If the rectangle at F2 above is bowed at its top and bottom (see Figure 11 d & e) the logarithmic curvature of the crystal is incorrect. A set screw (28) can be loosened and slide (27) when moved slightly will eliminate this bowing of the image.

Once these adjustments are made and the image is as close to a rectangle as possible, the monochromator crystal need not be adjusted again as long as the same radiation type is used. If all adjustments are correct (i.e. final spot focus adjustments) the reflex wheel should be turned until the image at F2 becomes triangular as the toothed screen (34) begins to cut into the main beam image.

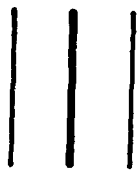
TYPES OF IMAGES OF THE MAIN BEAM WHICH CAN BE SEEN ON
A FLUORESCENT SCREEN HELD ~10 CM BEHIND THE CAMERA (F2)



A - QUARTZ CRYSTAL CORRECTLY HELD
B - CURVATURE OF QUARTZ CRYSTAL NOT SUFFICIENT
C - QUARTZ CRYSTAL BENT TOO STRONGLY
D - INCORRECT LOGARITHMIC CURVATURE, MOVE SLIDE TO RIGHT
E - INCORRECT LOGARITHMIC CURVATURE, MOVE SLIDE TO LEFT

Figure 11. <u>Types of Main Beam Images.</u>

SINGLE SIDED EMULSION
FILM IMAGE



DOUBLE SIDED EMULSION
FILM IMAGE

OBLIQUE VIEW OF DOUBLE
SIDED EMULSION FILM IMAGE

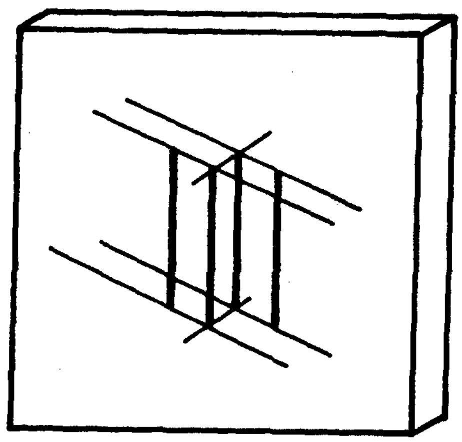


Figure 12. Main Beam Kα Doublet Splitting.

6. OPERATION

Once the instrument is aligned a wide angle x-ray diffraction pattern can be recorded photographically as a function of time or several patterns may be recorded statically on the same piece of film. Separate exposures at different temperatures may then be collected or a continuous change in the sample's WAXD pattern may be obtained as a function of temperature.

The x-ray generator, furnace cooling water (12), and control box power must all be on before beginning a run. Once all runs are complete these may be turned off.

6.1 Temperature Control

The starting and finishing temperatures are selected at the control box (see Figure 13). The Low Temp Control Variac (36) sets the temperature at the lower end of the desired range just as the High Temp Control Variac (37) sets the upper end of the range. Figure 14 (from reference 1) is the "setting selection chart". The higher temperature is selected first and its variac set, then the lower temperature is set as a percent of the higher temperature. For example, if you wanted to set the temperature range from 400° to $1000^{\circ}C$, first the setting of 60% would be dialed at the higher control and then 50% at the lower control ($400^{\circ}C$ is a setting of 30% on the higher control which is one half of 60%). If zero is set for the lower end of the range then the lower temperature is ambient. The Ranger Indicator Variac (38) is a motor driven variac which indicates the differential voltage between the high and low temperature variacs.

The starting temperature may be the lower or higher temperature by the selecting "up" or "down" respectively on the Temp direction switch (39). In either case the temperature will be raised to the starting temperature before the timer begins. When the end temperature switch (42) is in the "cyclus" position the power to the furnace will be shut off at the end of the experiment, when it is in the "continuals"

position the final temperature is maintained until new settings are made or the equipment shut down.

6.2 Time and Speed Selection

The length of time desired for the furnace to heat (or cool) the sample from the starting temperature to the final temperature is selected at the Time select switch (40). Discreet times of 0, 3, 4, 6, 12, 30, 40, 60, and 120 hours are available.

The speed of the film cassette must also be set. Speeds of 0.0, 0.5, 1.0, 1.5, 2.0, 5.0, 10.0, 15.0, and 20.0 mm/hour can be selected at the Film speed switch (41). The zero speed is used for static exposures. The speed selected is dependent on the length of the experiment and the diffraction characteristics of the sample.

6.3 Sample Preparation

The sample can be placed in any holder that fits the frame (10 mm x 5 mm). A metal frame is available commercially as well as a metal screen in a frame that powdered samples may be pressed into. Care must be taken not to press too hard since these frames are made of a deformable platinum-iridium alloy. In Figure 4 a solid polymer film is shown while in the Appendix a film sample was wrapped around the commercial frame that fits into the sample holder (5).

The sample can be mounted in the sample holder by either removing the entire holder or by inserting the frames through the sample access port (7).

Once the sample chamber is sealed, the environment can be set. The sample can be run in air, vacuum, or with a sweep gas by connecting the appropriate hoses to the Vacuum port (4).

6.4 Recording Film

Any type of x-ray sensitive film may be used. The size of the film should be 174 mm wide and any length up to 260 mm. The film must be loaded into the black paper film cassette in a darkroom and the cassette loaded in its holder (9). The film should be wide enough so that one edge is in the holder lip on the main beam side and extend out to capture the entire region of interest. The length of the film should be long enough to cover the entire experiment, depending on the time and film speed.

Once the the temperatures have been set, the time and film speed input, the sample mounted, and the film is in place the experiment can begin. At selected points in the experiment, the film position can be marked by moving the marking diaphragm (31) into position and momentarily opening the main beam catcher (8). The marking diaphragm is normally left out of position to avoid scatter and to allow the film to obtain the lower angle region of the pattern.

Once exposed the film should be developed promptly according to the standard procedures for that film. It can then be analyzed like any cylindrical film remembering that every two millimeters of film corresponds to 1 degree two theta. Several programs are available for this analysis [4 & 5].

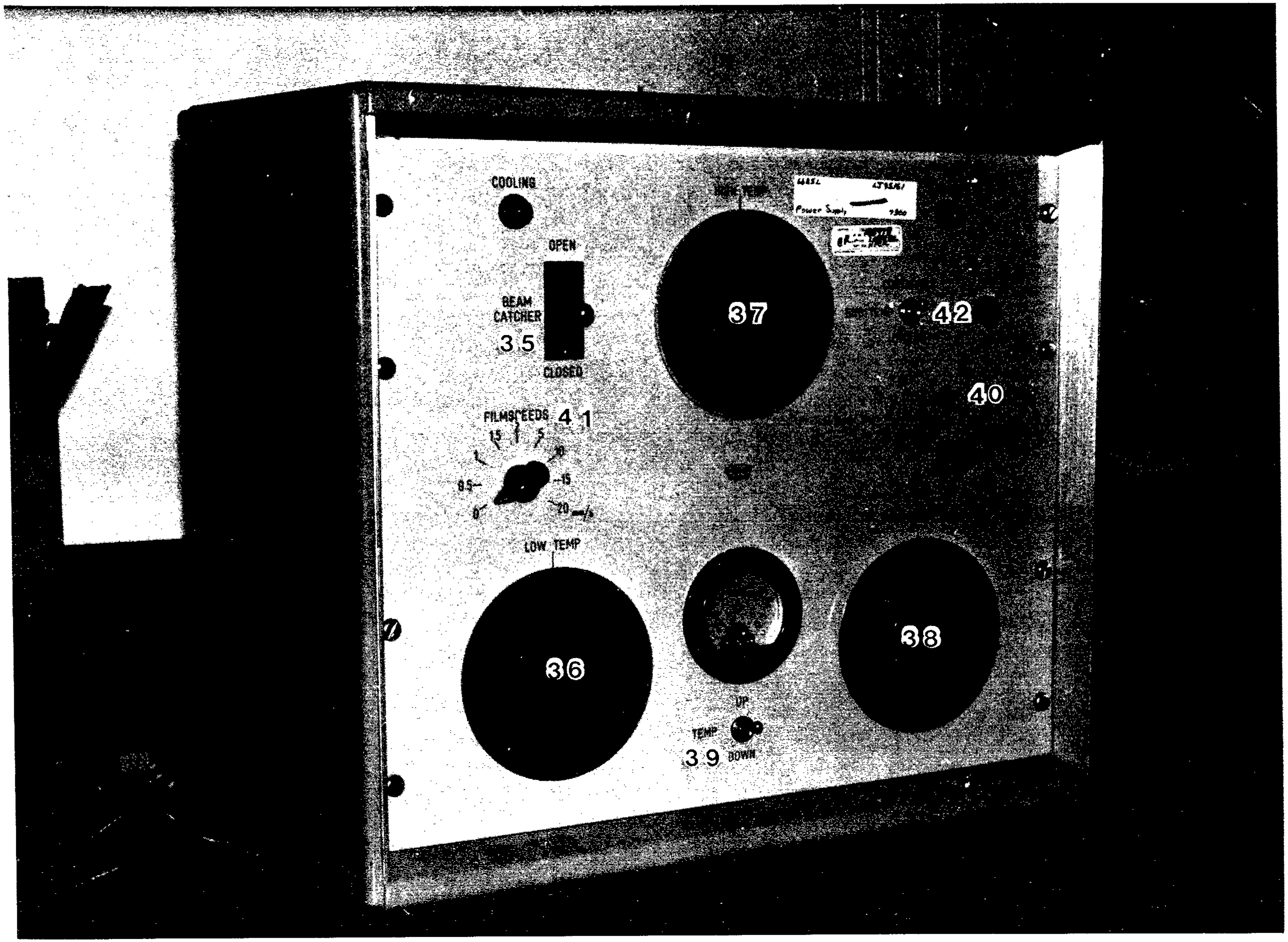


Figure 13. Control Box.

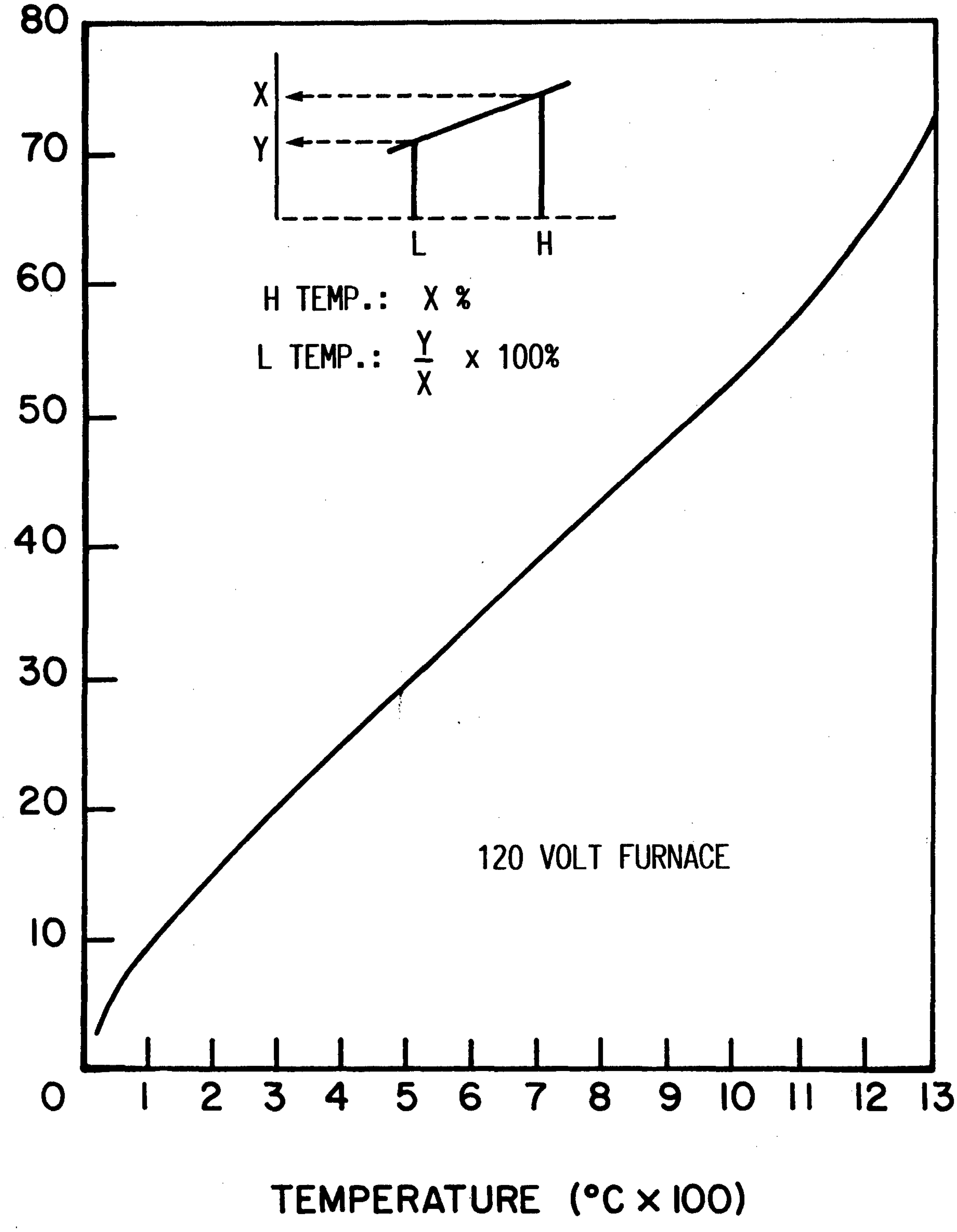


Figure 14. Temperature Control Chart.

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APPENDIX A

SAMPLE OPERATION RESULTS

Heat treatment is a commonly used technique for enhancing the mechanical properties of polymeric fibers. This is of particular interest to the Air Force in the ordered polymers area where fibers of Poly(p-phenylenebenzobisthiazole) (PBT) have been found to dramatically change on heat treatment [6]. This fiber was therefore chosen to test the initial operation of the G-L camera described in the main text of this report.

In this example the Guinier-Lenne camera was aligned according to the instructions in this report and a sample was made of as-cast PBT film wrapped around the open Pt-Ir frame. The film was oriented with its machine direction (direction of chain orientation) vertical such that the G-L camera would record the equatorial reflections of the fiber (the G-L diffraction plane is horizontal).

The conditions of the experiment were to monitor the WAXD pattern as the temperature was increased from ambient to near 600°C and then back to ambient in air. The low temperature variac was set at 0%, the high temperature variac was set at 35%, the end temperature switch set to "cyclus", and no vacuum or sweep gas was used. The temperature of the sample holder was periodically checked with the built-in thermocouple and a second thermocouple attached to the sample holder frame; both thermocouples registered the same temperature within a few degrees. Figure A1 shows a plot of the temperature versus time during this experiment.

A film speed of 15 mm/hr was selected to give an effective exposure time of 4 minutes through the 1 mm wide diffraction slit. The time of the experiment was set at 3 hours to get an average temperature increase of 4°C/min. The film cassette was loaded with a fast Kodak DEF-5 x-ray film to get a WAXD scan with as much intensity as possible. Figure A2 is a print of the film after exposure; main beam marks were placed at various times as reference points.

The optical density of the film negative was digitized and analyzed by program "PHOTO" [4]. The relative intensities plotted against Bragg angle at several times are shown in Figure A3.

From these and other similar plots one can readily observe several changes during the heating and cooling of this sample. The positions of the two peaks (e_1 and e_2) move to lower angles with increased temperature corresponding to larger dimensions, due to thermal expansion in the crystal since this change is reversible and occurs in heat treated samples as well (example not shown). More important is the dramatic increase in peak heights and narrowing of the peaks which occurs around 300^{o}C (see scans b, c, & d in Figure A3). The temperature of this transition which is observed only when heating as-spun PBT, occurs in the temperature range reported for a nonreversible second order transition seen by DSC [7]. The annealing of the sample to form more and larger crystals with greater degrees of perfection is a logical explanation for this transition and has been suggested from other studies [6].

One can conclude from this example [8] that crystal phase transitions of even weakly scattering organic materials can be observed by the Guinier-Lenne high temperature camera. Changes such as thermal expansion are even easier to detect.

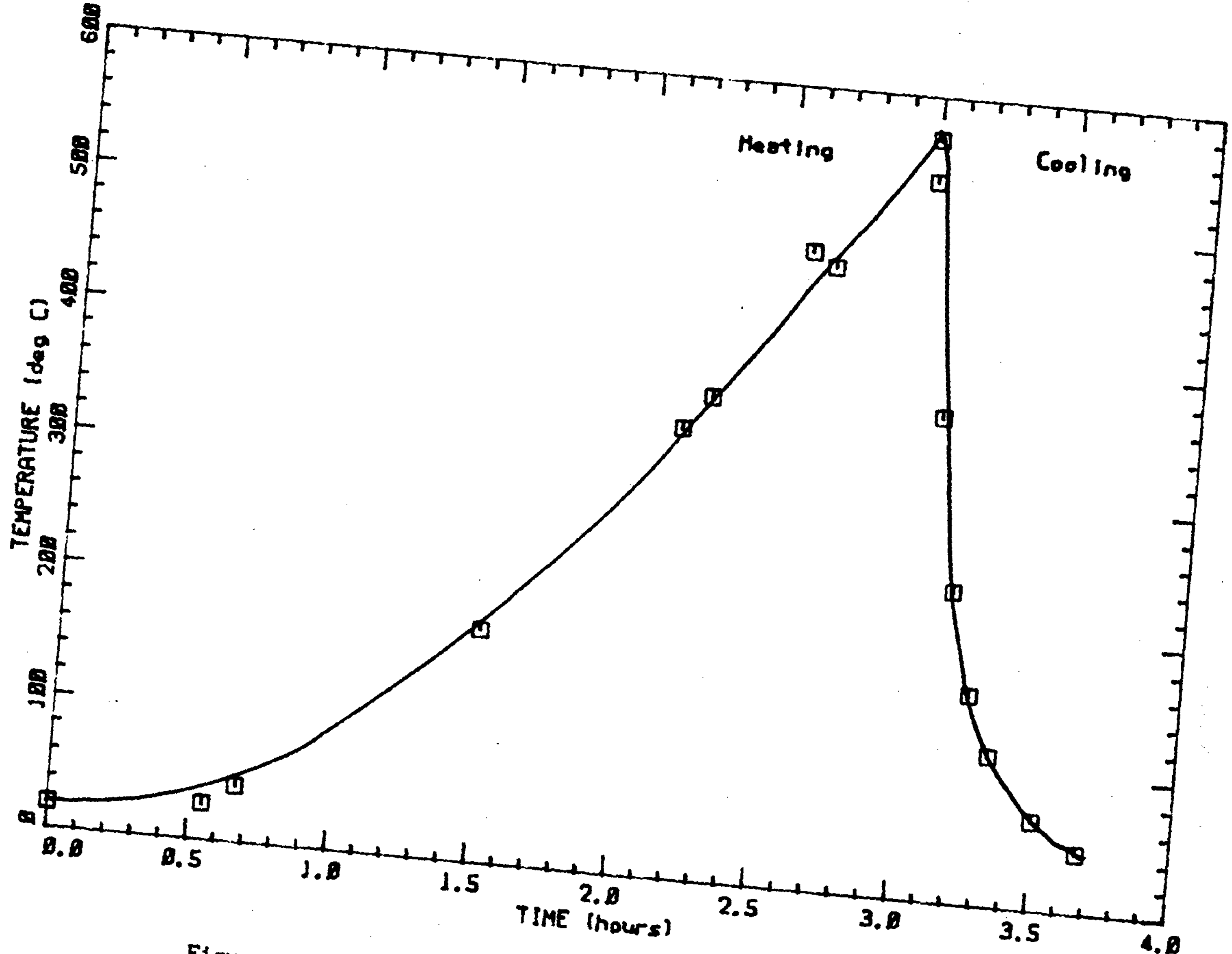

Figure A1. Time-Temperature Response of Guinier-Lenne Furnace.

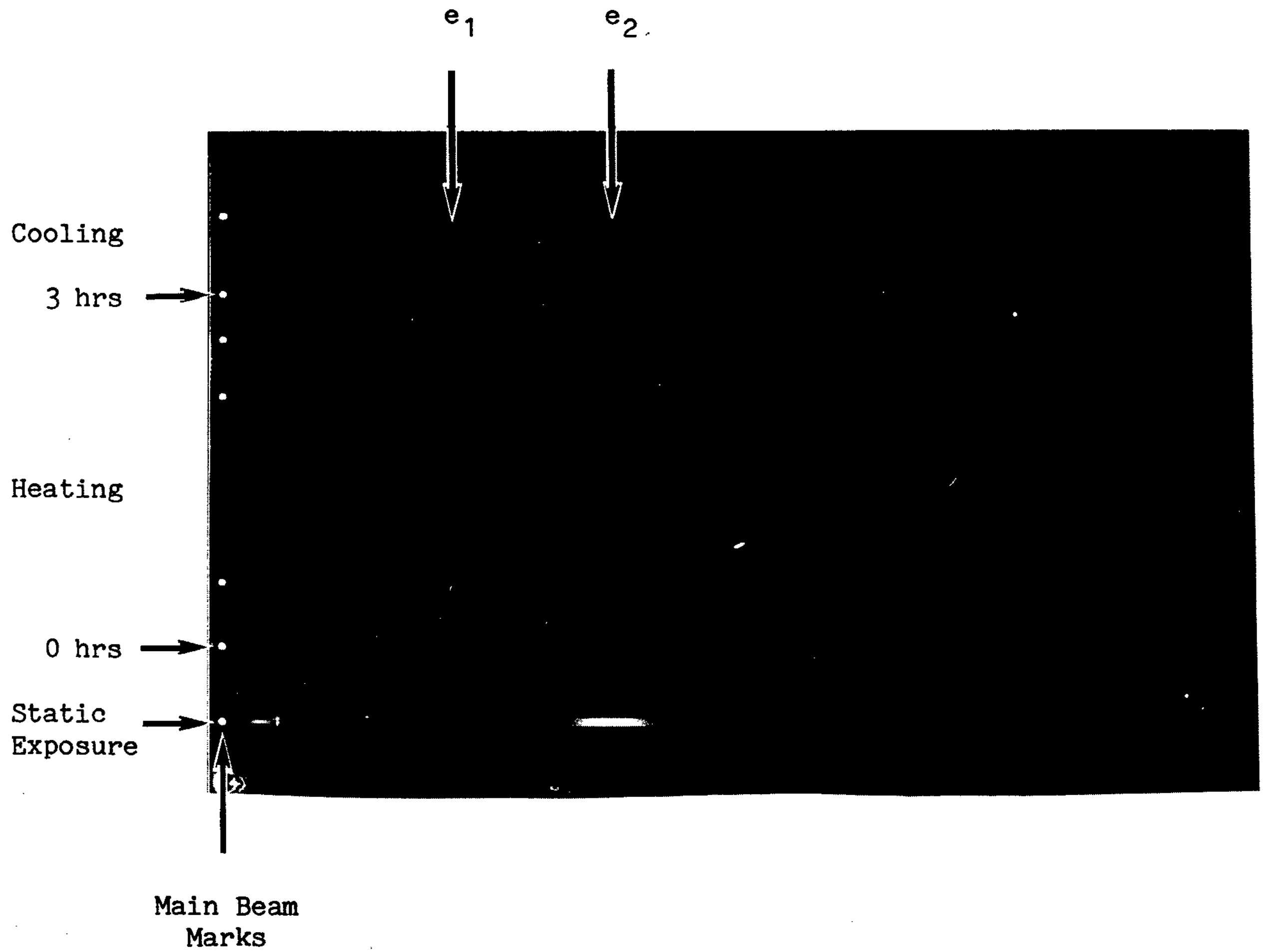

Figure A2. Guinier-Lenne Dynamic Diffraction Negative for PBT.

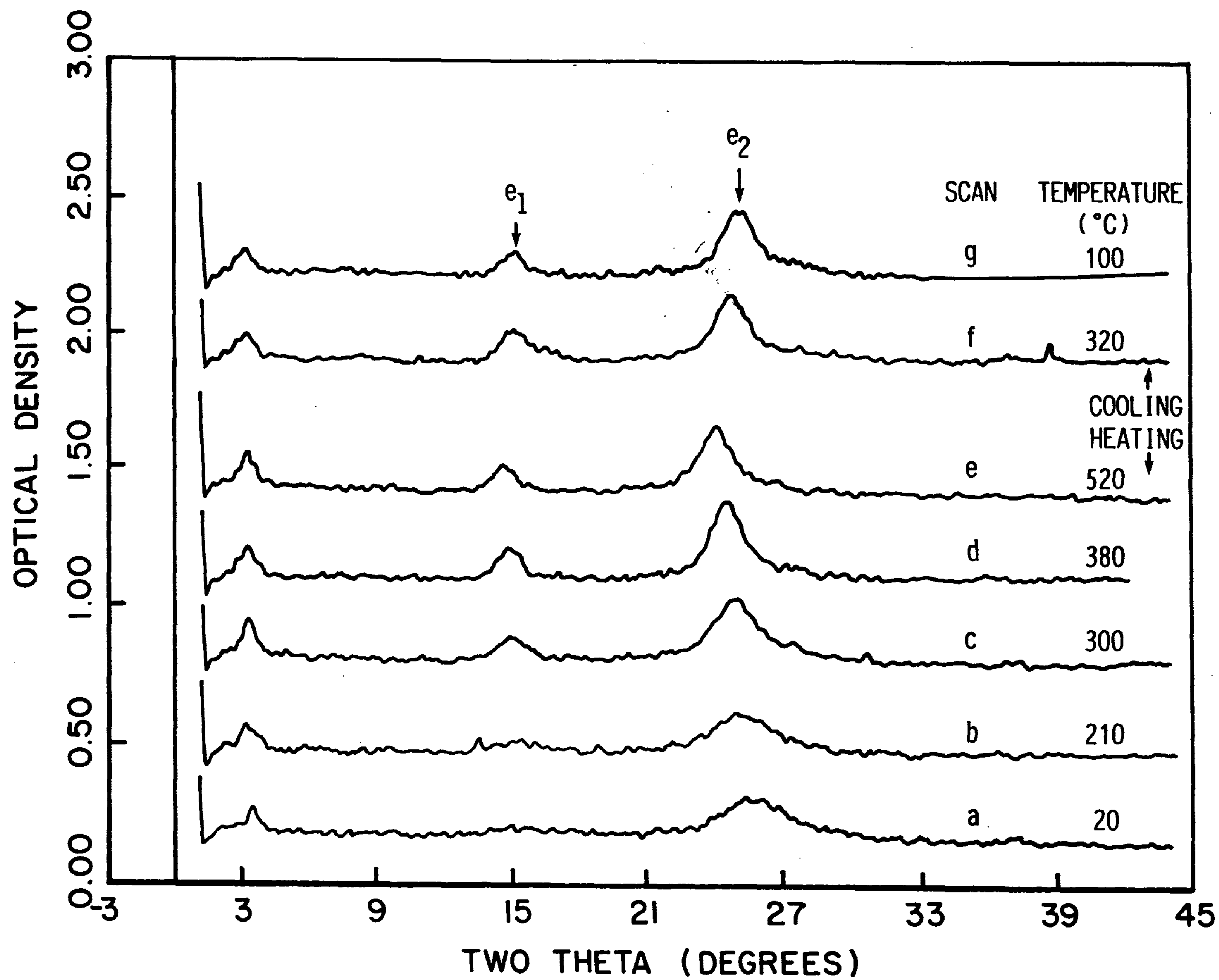

Figure A3. Selected Bragg Scans from PBT heating.

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